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Spin-echo NMR of ^{159}Tb in a single crystal of TbNi$_5$

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The field dependence of the hyperfine splittings of ^{159}Tb in a single crystal of TbNi$_5$ has been studied at liquid helium temperature by spin-echo NMR. The field, up to 8 tesla, was applied along the direction of easy magnetization of the crystal. Our measurements are in almost perfect agreement with computation based on the mean values of the crystal field parameters available in the literature for TbNi$_5$.

I. INTRODUCTION

The magnetic properties of the rare-earth cubic Laves phase intermetallic compounds have been the subject of extensive studies because of the high symmetry these compounds possess and, as a result, the small number of parameters required to describe their physical properties. A large body of data has therefore been accumulated about these compounds for both single crystals and polycrystals. In contrast to the cubic Laves phases compounds, far fewer studies have been made in compounds with lower symmetries. The RNi$_5$ series is the next natural candidate on account of its simple crystal structure, a hexagonal structure with one lanthanide ion per primitive cell. This series is particularly suitable for the study of exchange and crystal field interactions because, although there is a small contribution from the 3d band of nickel to the magnetization, Ni is itself nonmagnetic. The magnetic properties of these compounds therefore arise almost entirely from the incomplete 4f shell of the rare-earth ions. Furthermore, all the compounds in the series are congruent melting, excellent single crystals can be grown quite readily.

Detailed studies have been made of single crystals of the RNi$_5$ series by means of magnetization and susceptibility,[1–3] resistivity,[3,4] torque magnetometry,[5] high field magnetization measurements,[6] and polarized neutron scattering.[1,7,8] So far, however, no resonance studies have been made of the compounds in this series. Although Dalmas *et al.*[9] have investigated the RNi$_5$ (R =La, Gd, and Tb), compounds by μsr, in this technique, the muon enters the compound interstitially and cannot probe the field at the site of the lanthanide ion as does NMR. The majority of NMR measurements so far carried out on rare-earth alloys and compounds have been on polycrystalline samples and in zero fields. These measurements, although simple and straightforward, cannot give precise information because of the presence domain walls. The application of an external field to drive out the domain walls can cause severe inhomogeneous broadening, particularly in a strongly anisotropic system such as TbNi$_5$ which requires a large field to achieve this. In an oriented single crystal this is not the case and hence one can perform precise measurements on a single domain specimen.

In this paper we report the spin-echo NMR of ^{159}Tb in a single crystal of TbNi$_5$ in a field up to 8 T applied along the a axis of the hexagonal structure which is the direction of spontaneous magnetization.

II. EXPERIMENTAL PROCEDURE

The sample used in this study was cut from a master crystal, grown by the Czochralski method in a cold crucible induction furnace. The resulting crystal was oriented to better than 1° using the Laue back-reflection method, and a specimen having dimensions $1.8\times1.8\times5.3$ mm was cut with its long axis coinciding with the direction of easy magnetization of the crystal. This sample was first investigated by means of resistivity and low field magnetic measurements and was subsequently used in the present NMR experiment.

The crystal was incorporated as the central conductor of a specially designed coaxial resonator tunable from 2 to 7 GHz.[10] The resonator was located at the center of a superconducting solenoid whose axis coincided with the a axis of the crystal. The maximum deviation of the direction of the applied field from the crystal a axis is estimated to be less than 1°. Our computations show that such a small misalignment has no significant effect in the highly anisotropic TbNi$_5$ compound. The measurements were made at liquid helium temperature (4.2 K).

III. THEORY

The model employed here is that used by Waind *et al.*,[11] Ross *et al.*,[12] Prakash *et al.*,[13] and McMorrow *et al.*[14] The hyperfine interaction of the ^{159}Tb nucleus ($I=3/2$) with electrons of the parent Tb^{3+} ion ($J=6$) is treated as a perturbation on the electronic ground state.

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A. Electronic Hamiltonian

The electronic Hamiltonian representing the interaction of a given Tb^{3+} ion with its environment is written as

$$\mathcal{H}_{\text{el}} = \mathcal{H}_{\text{cf}} + \mathcal{H}_z \ . \tag{1}$$

At the site of hexagonal symmetry the crystal field Hamiltonian H_{cf} is

$$\mathcal{H}_{\text{cf}} = B_2^0 O_2^0 + B_4^0 O_4^0 + B_6^0 O_6^0 + B_6^6 O_6^6 \ , \tag{2}$$

where the O_m^n are the Stevens equivalent operators[15] and the B_m^n are the crystal field parameters. These parameters have been determined by Nait-Saada,[1] Gignoux and Rhyne,[7] and Goremychkin *et al.*[8] and are given in Table I. The data of Nait-Saada, and Gignoux and Rhyne are obtained from magnetization and susceptibility measurements and from inelastic neutron scattering studies of a single crystal of TbNi$_5$, respectively. Those of Goremychkin *et al.* refer to an inelastic neutron scattering study on a polycrystalline TbNi$_5$ sample. The agreement between the values of B_2^0 and B_6^6 obtained by these authors is excellent but there are significant differences in the values of B_4^0 and B_6^0.

The second term in the electronic Hamiltonian represents the Heisenberg exchange interaction and the Zeeman interaction between the terbium moment and the field B_0 at the terbium site. Thus we write

$$\mathcal{H}_z = g_j \mu_B (\mathbf{B}_0 + n g_J \mu_B \langle \mathbf{J} \rangle) \cdot \mathbf{J} \ . \tag{3}$$

B_0 includes the applied field B_a (after corrections for the demagnetizing field) and the dipolar field B_{dip} acting on the ion; n is the total isotropic bilinear exchange parameter given by

$$n = 3k_B \Theta_p / (g_J \mu_B)^2 J(J+1) \ . \tag{4}$$

Numerical values for TbNi$_5$ are given in Table II.

Although in the RNi$_5$ series there is no moment at the nickel site, both B_0 and n are enhanced by the susceptibility of the nickel's $3d$ electrons in the conduction band. This contribution of the nickel to the magnetization is more important at high temperatures where the $4f$ contribution is small but it is still significant at 4.2 K. The nickel's contribution is taken into account in our model by writing

$$\mathbf{B}_0 = (1+\alpha')\mathbf{B}_i, \quad n = n_{RR} + \alpha' n_{R\text{Ni}} \ , \tag{5}$$

where B_i is the applied field after correction for the demagnetizing field, $n_{R\text{Ni}}$ and n_{RR} are respectively the molecular field coefficients for the rare-earth nickel interaction and for the rare-earth rare-earth interaction and $\alpha' = \chi_{\text{Ni}} n_{R\text{Ni}}; \chi_{\text{Ni}}$ is the exchange enhanced susceptibility of the nickel (see Nait-Saada[1] for more details). For TbNi$_5$ Nait-Saada finds $\alpha' = -0.06$

TABLE I. The crystal field parameter B_n^m for Tb^{3+} in TbNi$_5$. The units are kelvins. (a) Saada (Ref. 2). (b) Goremychkin *et al.* (Ref. 8); (c) Gignoux and Rhyne (Ref. 7); (d) is the average of (a), (b), and (c) used in this work.

	(a)	(b)	(c)	(d)
B_2^0	3.84	3.67	3.84	3.84
$B_4^0 \times 10^2$	−0.04	−0.18	−0.24	−0.15
$B_6^0 \times 10^4$	−0.40	−0.12	−0.06	−0.19
$B_6^6 \times 10^4$	−4.0	−3.67	−3.67	−4.78

TABLE II. Magnetic properties of TbNi$_5$ in zero field. The data in the table are taken from Nait-Saada (Ref. 1).

T_c (K)	Θ_P (K)	χ_{Ni} emu/mol	α'	n (T/μ_B)	μ_s (μ_B/f.u.)	B_m (T)
23	17	50.2×10^{-4}	−0.06	0.81	7.6	6.34

For computational purposes it is more convenient to write $\mathcal{H}_z$ as

$$\mathcal{H}_z = -\boldsymbol{\alpha} \cdot \mathbf{J} \ , \tag{6}$$

where

$$\boldsymbol{\alpha} = \boldsymbol{\alpha}_{\text{ex}} + g_J \mu_B (1+\alpha')[\mathbf{B}_i + \mathbf{B}_{\text{dip}}] \ . \tag{7}$$

In terms of the exchange field b_{ex} acting on the projected spin $\sigma = (g_J - 1)J$ of Tb^{3+}, α_{ex} is given by

$$\boldsymbol{\alpha}_{\text{ex}} = (g_J - 1)\mathbf{b}_{\text{ex}} \ . \tag{8}$$

The exchange field is related to the equivalent molecular field acting on the terbium moment by

$$\mathbf{B}_m = -(g_J - 1)\mathbf{b}_{\text{ex}} / g_J \mu_B \ . \tag{9}$$

In the molecular field model b_{ex} is given by

$$\mathbf{b}_{\text{ex}} = \Gamma \langle \sigma \rangle = (3k_B \Theta_p / G) \langle \sigma \rangle \ , \tag{10}$$

where $G = (g_J - 1)^2 J(J+1)$ is the deGennes factor.

With $\sigma = (g_J - 1)\langle J_Z \rangle = 2.63$ (see below), $\Theta_p = 17$ K, and $G = 10.5$ we obtain $B_m = 6.34$ T for TbNi$_5$. An applied field of 8 T, the highest field used in our experiment, is therefore a significant contribution to the Zeeman term in $\mathcal{H}_{\text{el}}$.

The demagnetizing field $\mathbf{B}_{\text{dm}}$ is calculated by approximating our sample to a cylinder of the same cross section and length. We estimate the demagnetizing factor N to be -0.07. Therefore $\mathbf{B}_{\text{dm}} = \mu_0 N M_s = 0.08$ T at saturation which is not significant in the present work. (M_s was computed from the measurements of Nait-Saada[1] who gives $\mu_s = 7.6\mu_B$/f.u.) The dipolar field $\mathbf{B}_{\text{dip}}$ is the sum of the Lorentz field $\mathbf{B}_L$ and the field from the dipoles within the Lorentz sphere $\mathbf{B}_{\text{LS}}$. For TbNi$_5$ we find $B_L = \mu_0 M_s / 3 = 0.36$ T and $B_{\text{LS}} = -0.1$ T at saturation.

B. Hyperfine interaction

The theory of the hyperfine interaction in rare-earth alloys and compounds has been described in detail by McCausland and Mackenzie[16] and Waind *et al.*[11] We give below an outline of the theory and refer the reader to the above references for more details.

The hyperfine splitting of the electronic ground state of the rare ion in the solid is described by the following effective Hamiltonian:

$$\mathcal{H}_N = h\,[a_t I_z + P_t(I_z^2 - \mathbf{I}^2/3) + wI_z^3]\ , \qquad (11)$$

where h is the Planck constant, $\mathbf{I}$ the nuclear spin operator, and the z axis is taken along the total ionic moment $\langle \mathbf{J} \rangle$ which, in the present case, is along the crystallographic a axis. The parameters a_t, P_t, and w are respectively the total dipolar, total quadrupolar, and pseudo-octupolar parameters.

The dominant contributions to a_t and P_t come from the intra-ionic terms. These are given by

$$a' = a_0 \langle J_z \rangle / J + \text{2nd order term}\ , \qquad (12a)$$

$$P' = P_0 \langle 3J_z^2 - \mathbf{J}^2 \rangle / J(2J-1) + \text{2nd order term}\ , \qquad (12b)$$

where a_0' and P_0' are the free-ion coupling constants. In his review article Bleaney[17] gives $a_0' = (3180 \pm 30)$ MHz and $P_0' = (386 \pm 20)$ MHz for the terbium ion. Pelletier-Allard[18] have obtained $a_0' = (3168 \pm 18)$ MHz and $P_0' = (360 \pm 8)$ MHz from high resolution optical spectroscopy of Tb^{3+} in $\mathrm{TbCl_3}$. The second-order contributions are, in the present case where the electronic ground state is well isolated, small, about 1 MHz for a' and 3 MHz for P'.

The extra-ionic contribution a'' to the dipolar parameter is small, at the most 2% of the total; it is expressed in terms of an effective field $\mathbf{B}'' = (2\pi/\gamma)a''$ where $\gamma/2\pi = (10.13 \pm 0.02)$ MHz/T for ^{159}Tb.[17] Since the measurements were made in fields applied along the easy axis, all the contributions to $\mathbf{B}''$ are expected to be collinear with $\langle \mathbf{J} \rangle$ and hence with the intra-ionic field $\mathbf{B}'$. The total dipole parameter a_t may therefore be expressed as the algebraic sum of the intra-ionic and extra-ionic terms a' and a'', respectively.

We write the extra-ionic dipolar field as

$$\mathbf{B}'' = \mathbf{B}_i + \mathbf{B}_{\mathrm{dip}} + \mathbf{B}_{\mathrm{ce}}\ . \qquad (13)$$

$\mathbf{B}_i$ and $\mathbf{B}_{\mathrm{dip}}$ have been defined in the previous paragraph. $\mathbf{B}_{\mathrm{ce}}$ is the conduction electron contribution to $\mathbf{B}''$; it consists mainly of two terms. One arising from the polarization of the conduction electrons by the parent ion, here Tb^{3+}, which we write as $\mathbf{B}_P = \mathcal{K}_p \langle \sigma_P \rangle$. The other term is due to the polarization of the conduction electrons by the remaining ions in the crystal. This we take to be given by $\mathbf{B}_N = \mathcal{K}_N \langle \sigma \rangle$. The phenomenological coefficients $\mathcal{K}_N$ and $\mathcal{K}_P$ are assumed to be constant in a given series. In the present case $\langle \sigma_P \rangle = \langle \sigma \rangle = (g_J - 1)\langle \mathbf{J} \rangle$, the projected spin for Tb^{3+}. Therefore

$$\begin{aligned} \mathbf{B}_{\mathrm{ce}} = \mathbf{B}_P + \mathbf{B}_N &= -(\mathcal{K}_N + \mathcal{K}_P)\langle \sigma \rangle \\ &= -(\mathcal{K}_N + \mathcal{K}_P)(g_J - 1)\langle J \rangle\ . \end{aligned} \qquad (14)$$

Following McCausland and Mackenzie,[16] the hyperfine field is taken to be positive if it increases with the applied field, that is, parallel to the ionic moment $\langle \boldsymbol{\mu} \rangle$ and therefore antiparallel to $\langle \mathbf{J} \rangle$; hence the negative sign in the above equation. There is also a contribution to $\mathbf{B}_{\mathrm{ce}}$ arising from the oribital motion of non-s-conduction electrons in the vicinity of the ion. There are no measurements of the oribital contribution in the $R\mathrm{Ni_5}$ series, we therefore assume that this contribution is also collinear with $\langle \mathbf{J} \rangle$ and that it is included in the phenomenological parameter $(\mathcal{K}_N + \mathcal{K}_P)$.

From Mossbauer effect measurements in $\mathrm{GdNi_5}$ van Steenwijk *et al.*[19] found that the total hyperfine field $\mathbf{B}_{\mathrm{hf}}$ at the nucleus of the gadolinium ion is $-(25.2 \pm 0.5)$ T. Assuming that there is no moment on nickel in this compound they write

$$\mathbf{B}_{\mathrm{hf}} = \mathbf{B}_{\mathrm{cop}} + \mathbf{B}_P + \mathbf{B}_N\ . \qquad (15)$$

For $\mathrm{Gd}^{3+}(g_J = 1.993)$, $\langle \sigma \rangle = 3.476$ and $B_{\mathrm{cop}} = -33.2$ T; with these values Eqs. (14) and (15) give $(\mathcal{K}_N + \mathcal{K}_P) = -(2.30 \pm 0.14)$. From the diagonalization of the electronic Hamiltonian for Tb^{3+} in $\mathrm{TbNi_5}$ with zero applied field we find $\langle J_Z \rangle = 5.26$ from which we calculate $\langle \sigma \rangle = \langle \sigma_Z \rangle = (g_J - 1)\langle J_Z \rangle = 2.63$ and hence $B_{\mathrm{CE}} = (6.05 \pm 0.37)$ T. Therefore, in zero applied field, $a'' = (61.3 \pm 2.6)$ MHz.

In the hcp structure, the extra-ionic contribution to the quadrupole parameter is given by[20]

$$hP'' = 3eQ_n\gamma_N[1/2(3\cos^2\theta - 1)V_{cc}]/4I(2I-1)\ , \qquad (16)$$

where Q_n is the nuclear quadrupole moment, γ_N is the nuclear antishielding factor, θ is the angle between the c axis (the principal axis of the electric field gradients tensor) and the magnetic moment (in the present case $\theta = 90°$), and V_{cc} is the electric field gradient. V_{cc} is related to the quadrupolar crystal field parameter B_2^0 by

$$B_2^0 = -(e/4)\langle J \| \alpha_2 \| J \rangle \langle r^2 \rangle \gamma_E V_{cc}\ , \qquad (17)$$

where γ_E is the electronic antishielding factor, $\langle r^2 \rangle = 2.302 \times 10^{-21}$ m^2 is the mean square radius of the $4f$ shell,[21] and the Stevens equivalent-operator coefficient $\langle J \| \alpha_2 \| J \rangle = -1.1988 \times 10^{-2}$ in intermediate coupling. Eliminating V_{cc} between Eqs. (16) and (17) one can, given a value for the ratio of antishielding factors γ_N/γ_E, calculate P''. We have no value for the ratio γ_N/γ_E, however, Bunbury *et al.*[22] found for Tb^{3+} in $\mathrm{Tb(OH)_3}$ $\gamma_N/\gamma_E = (104 \pm 20)$. Although this ratio is expected to be host dependent, γ_N is usually in the range between -60 and -100,[23] and γ_E in the range between 0.3 and 0.8. We take the ratio γ_N/γ_E to be 100 ± 50 and find $P'' = -(36+12)$ MHz.

IV. RESULTS AND DISCUSSION

A. Zero field

Figure 1 shows the NMR spectrum of ^{156}Tb in zero field. The spectrum is dominated by the signal from domain walls. The lines from the domain, indicated by the arrows in Fig. 1, are identified by observing the evolution of the lines in a magnetic field. As the field is applied, the amplitude of the strong peaks decreases and these peaks disappear at about 1 T. On the other hand, the amplitude of the small peaks increases steadily with the applied field and the peaks evolve into strong sharp

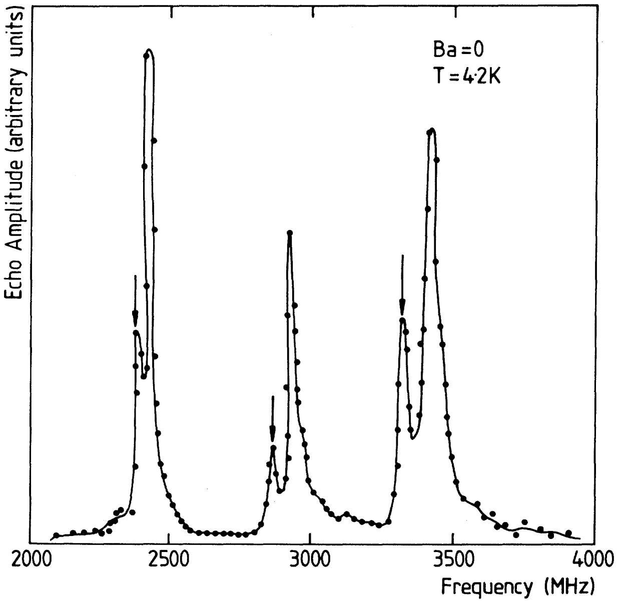

FIG. 1. The observed zero-field ^{159}Tb NMR spectrum in TbNi$_5$. The strong peaks belong to the spectrum from domain walls. The lines from the domain are indicated by the arrows.

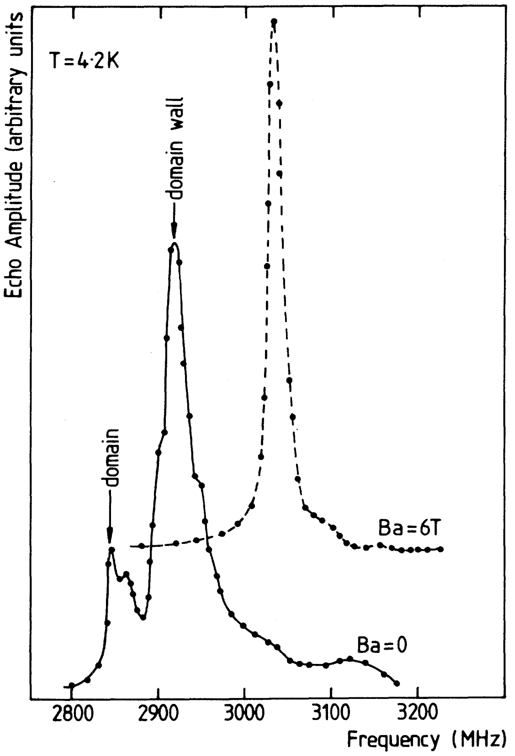

FIG. 2. The evolution of the central line of the ^{159}Tb NMR spectrum in TbNi$_5$. In zero field we observe a convolution of domain and domain-wall signals. In 6 T we observe a sharp line from a monodomain specimen.

lines at high fields. In Fig. 2 we show the central line in zero field and in a field of 6 T. It is not obvious why the frequency of the line from the domain wall is higher than that from the domain. All the data presented in this paper pertains to the signal from the domain.

The measured values for a_t and P_t in zero field are (2845 ± 14) MHz and (233 ± 7) MHz, respectively. The large uncertainties are mainly due to the difficulty in deconvoluting the domain spectrum from the complex zero field spectrum.

As we already stated, the total hyperfine parameters a_t and P_t are made up of intra-ionic and extra-ionic terms. The intra-ionic term is computed by diagonalizing the electronic Hamiltonian $\mathcal{H}_{\text{el}}$. This was carried out self-consistently. With the set of crystal field parameters in column (d) of Table I and value of B_m given in Table II, we obtained, at convergence, $\langle J_Z\rangle(=\langle J_a\rangle)=5.26$. Using this value in Eq. (12a) with Bleaney's free-ion coupling constants we find $a'=2790.4$ MHz which gives $a_t=a'+a''=(2852\pm4)$ MHz which is, within the experimental uncertainty, in agreement with our measurement. If we use the free ion coupling constants of Pelletier-Allard, we obtain $a_t=2842$ MHz. This is in excellent agreement with the measured value of 2845 MHz.

The measured and computed values of the quadrupole parameters are also in good agreement. Combining the value of $P'=281$ MHz (computed using Bleaney's P_0') with the estimated $P''=-(36\pm12)$ MHz gives $P_t=(245\pm12)$ MHz which is within two standard deviations from the measured value $P_t=(232\pm7)$ MHz. Using Pelletier-Allard's value for P_0' we obtain $P_t=226$ MHz which agrees, within the experimental uncertainty, with the measurement.

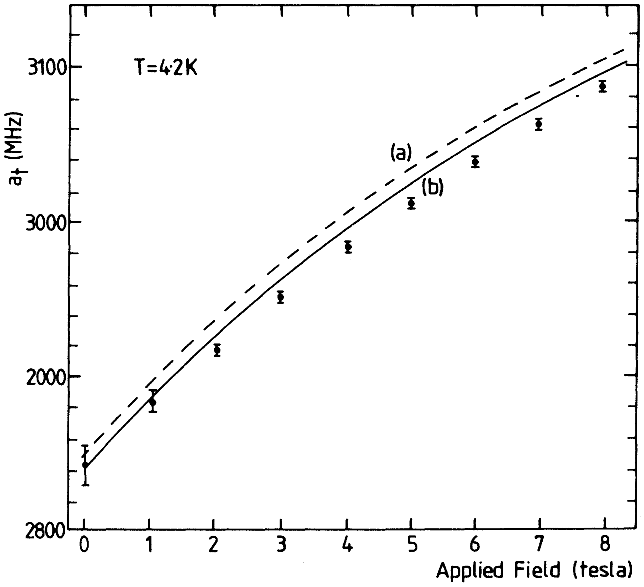

FIG. 3. The computed and measured field dependence of the dipolar hyperfine parameter a_t. The set of crystal field parameters given in column (d) of Table I are used for the computations. The solid line and the broken line are obtained using the hyperfine coupling constant given respectively by Pelletier-Allard and Bleaney.

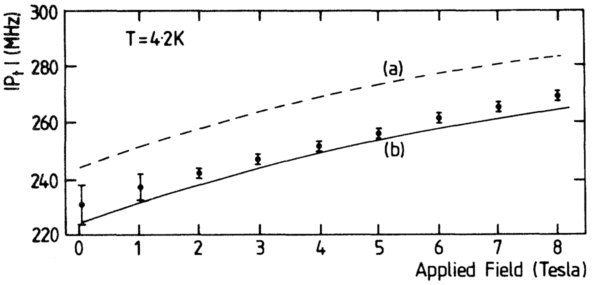

FIG. 4. The computed and measured field dependence of the quadrupolar hyperfine parameter P_t. The set of crystal field parameters given in column (d) of Table I are used for the computations. The solid line and the broken line are obtained using the hyperfine coupling constant given respectively by Pelletier-Allard and Bleaney.

B. Finite field

The variation of a_t and P_t with the applied field B_a is shown in Figs. 3 and 4. At high fields the NMR lines are sharp; the uncertainty on the measurement of a_t and P_t is 5 and 2.5 MHz, respectively. The two theoretical curves (a) and (b) are obtained using the free-ion hyperfine coupling constants of Bleaney and Pelletier-Allard, respectively. As can be seen in the figures, the agreement between the measured and computed field dependence of a_t and P_t better than 1% with the coupling constants of Pelletier-Allard, which is quite good considering that there are no free parameters in the calculations. The small discrepancy between computation and experiment could be attributed to small errors in the crystal field parameters and also, for the quadrupolar parameter P_t, to the uncertainty in the ratio of antishielding factors γ_N/γ_E.

V. SUMMARY AND CONCLUSIONS

We have measured the field dependence of the hyperfine splitting of ^{159}Tb in a single crystal of TbNi$_5$. We have interpreted our measurement using calculations based on the mean value of the crystal field parameters available in the literature and the magnetic measurements of Nait-Saada. The exceptionally good agreement we find between the computation and the measurements confirm that the sets of parameters used for the computation give a quite satisfactory description of the ground state of Tb^{3+} in TbNi$_5$.

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